

17

9011-1001

The crosslinker exemplified herein is a polyfunctional aziridine liquid crosslinker, such as, for example, 1-aziridinepropanoic acid, 2-methl-, 2 ethyl-2-(3-(2-methyl-1-WHAFIS-CLAIMED)IS) methyl)-1,3-propandiyl ester marketed by Zeneca Resin, Wilming of graft coated substrate; the substrate comprising polyethylene, and a graft coating covalently bonded therethy wherein that digraft coating comprises a monopolymer eactive carboxyl functionality, in both water-based and organic solvent 2 a The graft coated substrate of claim 1, wherein the graft coating comprises a polymer selected from the group consisting of a wrethane, and cooking comprises a polymer selected from the group consisting of a wrethane, and coating comprises agents materials selected from the group consisting of a pigment or colorant and fire retarding agent, and combinations thereofy curing agants or hardeners. 2.5., including those comprising trimeth hereign coated substrate of claim 4 wherein the substrate comprises a life Products polyethylene having addensity rangings from about 0.930 g cm<sup>-3</sup> to about 0.940 g cm<sup>-3</sup>.

5. The graft coated substrate of claim 1 that comprises a polyethylene having an The Grafting Solution and Process average molecular weight ranging from about 100,000 amu to at least 6 x 10<sup>6</sup> amu.

Parts A and B are mixed in a suitable proportion stirred to a uniform solution, and the 6. The graft coated substrate of claim 1, wherein the substrate comprises a resulting grafting solution is applied to the PE substrate to be treated. The time necessary for polyethylene selected from the group consisting of low density polyethylene, a linear low the reaction to run to completion depends up the reaction temperature, the reagents employed density polyethylene, a medium density polyethylene, a high density polyethylene, a high and the desired properties of the grafted PE. Generally, the solution is air dried onto the PE density, high molecular weight polyethylene, a high density, ultra high molecular weight substrate and then cured by the application of heat for a time period ranging, e.g., from about polyethylene, an ultra-high density polyethylene, and combinations thereof.

1 to about 4 hours, at a temperature ranging, e.g., from about 100 to about 150 degrees F. The graft coated substrate of claim I that is formed into an article of manufacture selected from the group consisting of a pipe or tube, a curved or planar sheet, a beam, a mojent temperature, e.g., 25-30 degrees C. for up to for more days: board, a rod or shaft, a container for solids or fluids, and combinations thereof.

8. The graft coated substrate of claim 7 wherein the pipe is selected from the group consisting of straight pipe, bent pipe, a straight pipe joint, an elbow joint, an end-cap, a heat-shrinkable joint, and combinations thereof.

time particle by exposure to a standardized source of intense radiant heat, as described in time particle by exposure to a standardized source of intense radiant heat, as described in greater detail in the Examples. Infra. Surface energy was tested using standardized wet-consisting of single wall pipe, pipe with a plurality of walls nested one within the other, pipe with a single insulating layer between two concentric walls, and pipe with a plurality of with a plurality of

concentric insulating layers.
Surface Energy Testing

10. The graft coated substrate of claim 1 that resists melting and burning for a time in A number of art-known methods are available is tested by exposure to a concentration of a time in the article is tested by exposure to a concentration of a time in the article is tested by exposure to a concentration of the surface of the concentration of the surface of the concentration of the surface of the concentration of the concentration of the surface of the concentration of the co

period ranging from about 1 to about 18 minutes, when the article is tested by exposure to a of adhesion of paints, cements, adhesives minutes, when the article is tested by exposure to a of planar heated surface that is heated to a temperature ranging from about 800 to about 960°C,

10

٠.

115

20

**25**<sup>5</sup>

35

30

30



and wherein the heating panel is a rectangle that measures about 25 x 51 cm, and the graft coated substrate is positioned at a distance of about 12.5 cm from the heating panel.

- 11. The graft coated substrate of claim 1 that has a surface energy ranging from about 113 raincles were instant the there are face change by a translation of surface 56 to about 80 dynes/cm<sup>2</sup>.
- 12. The graft coated substrate of claim 1 that has a surface energy of at least 80 of the graft daybear depend upon the surface charty. A communication available week dynes/cm<sup>2</sup>.

  dynes/cm<sup>2</sup>.

  Jakity includes the Caroline that Profite Real Politics and the Company, December 1991. The test
- 13. A process for modifying the surface of a solid polyethylene substrate, comprising covalently grafting a heat resistant coating onto said substrate by
- (a) applying to a solid polyethylene substrate, a liquid composition comprising effective amounts of a monomer, prepolymer, a graft initiator, a catalyst and a polymerization promoter, under conditions effective to promote grafting of the monomer or prepolymer to the solid polyethylene substrate to form a coating on the substate, and

  (b) curing the applied composition.
- M. The process of claim 13 wherein the monomer or prepolymer is selected from the appropriate the process of claim 13 wherein the monomer or prepolymer is selected from the group consisting of a vinyl monomer, a urethane monomer, an epoxy monomer, a siliconthe following the followi
- 15. The process of claim 13 wherein the graft initiator is a metal ion, present in an subject to receive to mitiate radical formation in the polyethylene substrate.
- 16. The process of claim 15 wherein the graft initiator is present in a concentration ranging from about 0.01 to about 1.0%, by weight.
- 17. The process of claim 15 wherein the graft initiator is selected from the group consisting of ions of iron, silver, cobalt, copper, cerium and combinations thereof.
- 18. The process of claim 13 wherein the catalyst is a peroxide present in the liquid composition in a concentration ranging from about 0.1 to about 5% by weight.
- 19. The process of claim 13 wherein the catalyst is an selected from the group consisting of benzoyl peroxide, methyl ethyl ketone peroxide, 1-butyl hydroperoxide and combinations thereof.
- 20. The process of claim 13 wherein the polymerization promoter is present in a concentration effective to react with, and crosslink, the monomer or prepolymer.
- 21. The process of claim 20 wherein the polymerization promoter is a polyfunctional aziridine liquid crosslinker.
- 22. The process of claim 13 wherein the substrate is a polyethylene having a density ranging from about 0.930 g cm<sup>-3</sup> to about 0.940 g cm<sup>-3</sup>.

10

11

5

15

1 %

20

25

30

28

## **PART B**

Crosslinker CX-100

1.8

9011-1001

Preparatog of the process of claim 13 wherein the liquid composition is applied to the substrate
by a method selected from the group consisting of brushing, dipping, spraying and
ancombinations thereof promers, catalyst, graft initiator system and the other ingredients of
the composition is self-curing.
The resulzing The process of claim 13 wherein the applied composition is cured by heating the
coated substrate at a temperature and for a duration sufficient to cure the applied coating.

26. The process of claim 25 wherein the applied composition is cured at a Preparation of the FORMULATION:

temperature ranging from about 60 to about 200 degrees F, for a time period ranging from The Part A solution was mixed into a separate container with the Part B solution, in about 30 minutes to about 6 days.

the weight proportions shown above by Table 1. The mixed formulation was stirred to a 27. The process of claim 13 wherein the liquid composition further comprises a uniform solution for the grafting process. compatible flame retardant agent.

Application of the process of claim 27 wherein the flame retardant agent is a phosphorous-based flame retardant agent is a phosphorous based flame retardant agent is a phosphorous spraying. 29 The process of claim 27 wherein the flame retardant agent is relected from the 1 group consisting of chlorinated phosphate esters melamine derivatives poligomeric phosphate festers bromonyl ether phosphate product, and phosphonates.

The process of claim 27 wherein the flame retardant is selected from the group consisting of dimethyl methylphosphonate, diethyl-N, N-bis (2-hydroxyethyl) aminomethyl phosphonate, oligomeric chloroalkyl phosphate/phosphonate, tri (1, 3-dichloroisopropyl) phosphate, oligomeric phosphonate, tributyl phosphate, isopropylated triphenyl phosphate water-Based Grafting For hunation with a repolytical ester, and combinations thereof.

Is 12cThe process of claim 30 wherein the flame retardant agent is dimethyl methylphosphonate, were also prepared. Table 2. be an provides a water-based urethane formula 32.2. The process of claim 13 wherein the liquid composition is first prepared without the polymerization promoter, and the process further comprises the step of mixing the polymerization promoter with the liquid composition prior to application of the liquid composition to the substrate polymer NeoRez 967.

33. The process of claim 13 wherein the liquid composition further comprises a polymer selected from the group consisting of a vinyl polymer, a urethane, an epoxy, a polysilicone and combinations thereof; wherein said polymer is suitable for grafting to the substrate.

(Witco Organo Silicones Group/OSi Specialties, Inc.)

34. A solid polyethylene substrate comprising a graft coating covalently bonded thereto, prepared by the process of claim 13.

20

25

20

5

10

10

15

15

25

30

35. An article of manufacture comprising a graft coating covalently bonded thereto, prepared by the process of claim 13.

36. A liquid composition for graft coating a solid polyethylene substrate with a coating that comprises at least one non-polyethylene polymer, comprising an effective amount of a monomer or prepolymer, a graft initiator, a catalyst and a polymerization Unitable projections Needles 9679 was taken in a confiner and is it were added promoter.

n n n....

That they have